

PATENT SPECIFICATION



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COMPLETE SPECIFICATION.

Improved Manufacture of Anhydrous Alkali Polysulphides.

We, I. G. FARBENINDUSTRIE AKTIEN-GESELLSCHAFT, a Joint Stock Company, organised according to the laws of Germany, of Frankfurt a/Main, Germany, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

10 Anhydrous alkali polysulphides have hitherto been made by an inconvenient and costly process, namely by the reaction of sulphur on a mono-sulphide or
15 ~~hydro-sulphide in alcoholic or ethereal solution.~~ Another process has consisted in heating hydrated monosulphide with an excess of sulphur in a current of hydrogen, the latter serving for removal of the water of the reaction and subsequent separation of the excess of sulphur in the form of sulphuretted hydrogen. Neither process has led to a definite sulphide, for instance tetra-
20 sulphide, in a sufficient degree of purity and commercial success has not attended either process.

According to this invention anhydrous, practically pure polysulphide containing any desired ratio of alkali to sulphur is
30 obtained in a simple manner by mixing molten sulphur with anhydrous alkali sulphide, made by reducing an alkali-sulphate by means of a gaseous reducing agent. The temperature of the fused
35 mass is raised gradually in correspondence with the rising melting points, the initial temperature being about 200° C; the vessel for the reaction is preferably a directly heated closed iron vessel provided with a stirrer. In order to avoid
40 loss of sulphur by combustion, the reaction is advantageously carried out in an indifferent atmosphere, for example in nitrogen. By suitably adjusting the dose
45 of the added alkali sulphide one can obtain a polysulphide of any desired composition, that is to say there is brought into the reaction a quantity of the constituent materials corresponding with the
50 different proportions necessary for the formation of the desired sulphide. As the reaction is exothermic and the sodium sulphide, for instance is very prone to

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react, it generally suffices to heat the mixture to the temperature of the reaction, whereupon the process proceeds without further application of heat and is completed by the spontaneous rise of temperature in the mass. When the mass has become homogeneous it is removed from the vessel and cooled with exclusion of air. In order to eliminate small quantities of impurities originating, for instance, from the sulphur used, the mass before it solidifies may be allowed to settle or filtered through a hot filter.

The following Examples illustrate the invention:

EXAMPLE 1.

For making sodium tetrasulphide, 350 kilos of lump sulphur are melted in a directly heated closed iron stirring vessel. When the temperature has risen to 180—200° C., there are introduced continuously 300 kilos of anhydrous sodium sulphide of 98 per cent. strength, which has been made by reduction of the sulphate by means of a suitable gas. The sodium sulphide dissolves in the sulphur immediately, notwithstanding its fragmentary form, with considerable evolution of heat and during the introduction the temperature rises without additional heating to 320—340° C. The temperature remains constantly above the melting point of the mixture. The homogeneous mass is now forced by pressure of nitrogen into iron drums in which it solidifies. The solidified mass is ground and contains about 45 per cent. of sodium sulphide and 55 per cent. of sulphur.

EXAMPLE 2.

For making sodium disulphide 42 parts of sulphur and 100 parts of sodium sulphide of 98 per cent. strength, which has been made by reduction of the sulphate by means of a suitable gas, are mixed and heated in the manner described in Example 1, the temperature being allowed to rise gradually to about 440° C. The homogeneous solidified mass contains 70 per cent. of sodium sulphide and 28.5 per cent. of sulphur.

EXAMPLE 3.

300 kilos of anhydrous sodium sulphide of 98 per cent. strength are removed

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from the furnace in which the material is being produced by gaseous reduction of the sulphate and introduced directly into the reaction vessel. To this mass, 5 heated to about 700° C, 350 kilos or fused sulphur are added. The heat remaining in the hot sodium sulphide and the heat of reaction which produces the polysulphide supply the heat necessary for fusion 10 of the mass undergoing reaction.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we 15 claim is:—

1. A manufacture of alkali polysulphides by the addition of sulphur to an alkali sulphide, wherein anhydrous

alkali sulphide produced by reduction of an alkali sulphate by means of a suitable 20 reducing gas is caused to react directly with molten sulphur in the proportion necessary for obtaining the desired polysulphide.

2. A manufacture as claimed in claim 1, wherein the sodium sulphide is caused 25 to react with the fused sulphur, or the fused mass rich in sulphur, under such conditions that the mass remains liquid during the reaction. 30

Dated this 26th day of July, 1932.

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